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SUITABILITY OF LONGLEAF PINE FOR PAPER PULP.

By HENRY E. SURFACE, *Chemical Engineer in Forest Products*, and ROBERT E. COOPER, *Chemist in Forest Products.*

SOUTHERN PINES FOR KRAFT PULP.

The southern pines have not, until within the last few years, been considered suitable for paper pulp. Their resinous nature is the chief drawback in most processes of paper making. The recent development in Europe, especially in Sweden and Norway, of the sulphate process, however, and the superior quality of the product made from resinous woods has turned attention to longleaf and other southern pines as a possible source of pulp in this country. These pines have long, thick-walled fibers, and also high specific gravities, implying large yields per cord, and therefore seem particularly adapted for the manufacture, at low cost, of strong wrapping papers. The waste wood from the lumber industry in the South suggests a source of cheap raw material.

While the sulphate process can be used in the manufacture of bleaching pulps, its principal product is an undercooked, nonbleaching, brown pulp known as "kraft" pulp, the term, a German one, signifying strength. True to its name, this pulp produces a remarkably strong paper, very resistant to wear.

Kraft papers, which may be made by the soda as well as by the sulphate process, are especially adapted for wrapping purposes. Wrapping papers stand third among the paper products of the United States, being exceeded in amount and value only by news and book papers. In 1909 the production of wrapping papers of all kinds aggregated 764,000 short tons, with a value of $42,296,000.[1] The value of wrapping papers imported in 1912 was $846,500.[2] Complete

[1] Tariff Board Report, Pulp and News Print Paper Industry, 1911, p. 21. Senate Doc. 31, 62d Cong., 1st sess.

[2] Bureau of Foreign and Domestic Commerce, Monthly Summary of Commerce and Finance for December, 1912, p. 744.

statistics for recent importations of kraft paper are not available, but in 1908, three years after its introduction into the United States, the imports amounted to between 10,000 and 12,000 tons.[1] In 1912 the imports of unbleached sulphate pulp from Sweden alone were approximately 21,600 short tons, and from Norway 8,400 short tons.[2]

Manila wrapping papers, including the better imitation manilas, have generally been considered the strongest and best wearing, but the light-weight kraft papers give the same service as manilas almost twice as heavy. Although strong, light-weight wrapping papers are made in this country from sulphite pulps, the imported kraft papers and papers made from imported kraft pulps have proved too formidable competitors for even the best wholly-domestic product of this kind. The immediate success and largely increasing use of kraft products has brought on the market imitations, colored to resemble the genuine, made from strong sulphite pulp or from such pulp together with ground, steamed-wood pulp. Although some of them are quite strong in the light weights, they are not equal to the genuine in other ways. The opportunity for developing an increased domestic output of kraft products from native woods is apparent.

The above-mentioned conditions led the Forest Service to conduct a series of tests at the Forest Products Laboratory, maintained in cooperation with the University of Wisconsin, Madison, Wis., in order (1) to determine the suitability of the southern pines for paper pulps; (2) to ascertain the effects of varying cooking conditions in the sulphate process of pulp making; (3) to compare the sulphate process with the soda process. Only longleaf pine has so far been used in the tests, of which this bulletin gives the results under such preliminary analyses as have been made at this time.

LUMBER WASTE AVAILABLE FOR PULP MAKING.

The total stand of longleaf pine (privately owned) was estimated by the Bureau of Corporations in 1910 at 232 billion feet board measure, while for all southern pines the amount was placed at 384 billion feet. The lumber cut from these pines in 1910 amounted to 14 billion feet. The sawed lumber represents approximately one-half the volume of the log as it comes to the mill. Bark and sawdust, which are valueless for paper making, constitute a large proportion of the waste, but it is safe to say that 20 per cent of the volume of the log, exclusive of the bark, is lost in slabs, edgings, and trimmings. Tops and defective logs left in the woods and small logs which at present are converted into lumber with little or no profit would furnish a supply of raw material for pulp making even greater than that derived from the mill waste.

[1] Pulp and Paper Investigation Hearings, 1909, Vol. V, p. 3041. House Doc. 1502, 60th Cong., 2d sess.

[2] From estimates made by the Swedish Wood Pulp Association in 1913 and furnished the Forest Service by Mr. M. Gintzler, New York City.

The waste wood mentioned is not as a rule the clean, clear material to which pulp mills have been accustomed. But when the soda and sulphate processes are employed, the presence of knots, pitch pockets and streaks, and remnants of decayed wood and bark are not very objectionable. The expense of handling and preparing slabs and other irregular sizes and shapes, however, is greater than for round pulpwood, so the initial cost of such material must be low enough to offset the extra cost incident to its use.

PULP MAKING PROCESSES APPLICABLE TO LONGLEAF PINE.

Four or five mills are at present using southern pine mill waste for the manufacture of wrapping paper and similar products, three of which employ the sulphate process. Several other sulphate mills are either projected or in course of construction. Because of the resinous nature of the wood the preparation of paper pulp from longleaf pine is confined to the soda and sulphate processes, unless special extraction treatments are employed preliminary to cooking.

The soda process consists in digesting suitably prepared wood with caustic soda (NaOH) solution. The cooking results in dissolving the lignin and resin constituents of the wood, and separating the individual fibers from one another. The action depends partly upon the direct solvent and saponifying power of the caustic soda, and partly upon the hydrolysis of the wood in the presence of water at high temperatures, forming organic acid products which unite with the alkali present. Cellulose, of which the fibers are chiefly composed, withstands the cooking action, except under very severe treatment.

The spent cooking liquor, or "black liquor," is separated from the pulp fibers and evaporated; the residue is calcined in a furnace, and the soda compounds are recovered as "black ash," an impure sodium carbonate (Na_2CO_3). This ash is dissolved in water, and the solution is causticized with freshly burned lime; the resulting caustic soda is again used in cooking. The losses of soda occurring in the operations are made up by adding fresh soda ash (commercial sodium carbonate) previous to causticizing.

The sulphate process is similar to the soda process, except that sodium sulphide (Na_2S) is employed as a cooking chemical in addition to the caustic soda. The sodium sulphide is derived from sodium sulphate (Na_2SO_4), which is added during the recovery operations to make up for the losses, and it is from this chemical that the process derives its name. The sodium sulphate is mixed with the black ash and subjected to a high temperature in a "smelter"; this treatment reduces it to sodium sulphide, although the reaction is not complete. The "smelt," containing sodium carbonate, sodium sulphide, and unreduced sodium sulphate, is dissolved in water and the solution is causticized, as in the soda process, with lime, which has, however,

little action on the sulphide and the sulphate. During cooking the organic acids produced react with the sodium sulphide[1] as well as with the caustic soda, so that in calcining both chemicals are recovered as sodium carbonate. If desired, soda ash may be added to the smelt solution before causticizing in order to increase the proportion of caustic soda in the cooking liquors. Some mills have also found it advantageous to mix with the causticized cooking liquors some of the black liquors diverted from the recovery operations.

The soda and sulphate processes can be applied to extracted or steam-distilled chips from which rosin and turpentine have been removed. Turpentine can also be obtained from resinous chips during the cooking operations by condensing the "relief" from the top of the digester. However, the turpentine is very impure, and in the case of the sulphate process contains organic sulphur compounds from which it is separated with great difficulty.

EXPERIMENTAL METHODS.

KINDS OF TESTS.

The tests made by the Forest Service were of two classes: (1) Autoclave tests and (2) semicommercial tests. The autoclave tests comprised several series of cooks made to determine the effects of varying the cooking conditions of the sulphate process. The semicommercial tests include cooks made by the soda as well as by the sulphate process. The semicommercial sulphate cooks employed such cooking conditions as the autoclave tests indicated would give good results, while the tests using the soda process were made with cooking conditions that would give results comparable to those obtained from the sulphate cooks. Because the semicommercial tests show in a more direct manner the possibilities of preparing paper pulp from longleaf pine, they will be discussed before the autoclave tests.

WOOD USED.

The test material consisted of longleaf pine (*Pinus palustris* Mill.) from two localities, Perry County, Miss. (shipment L–3), and Tangipahoa Parish, La. (shipment L–176). A portion of the former, consisting of edgings containing approximately equal amounts of sapwood and heartwood, was used for cooks 176–1, 2, and 3 of the semicommercial soda tests (Table 3), and another similar portion of the same shipment was used for cooks 1 to 65, inclusive, of the autoclave tests. The average bone-dry weight of the wood used in these autoclave tests was 30.4 pounds per cubic foot green volume; the maximum and minimum values were 36.4 and 26.6 pounds, respectively. The wood was fairly free from resin. The remaining cooks employed

[1] In this reaction volatile organic sulphur compounds having extremely disagreeable odors are produced. Unless these odors are eliminated, or held in check by proper means, sulphate pulp mills are highly objectionable except in sparsely populated regions.

two butt logs (15 and 22 inches diameter) of the Louisiana wood, including all of the sapwood and heartwood. These logs were quite resinous, but were free from knots. They had an average bone-dry weight of 35.5 pounds per cubic foot green volume. The maximum and minimum weights were 40.1 and 32.3 pounds, respectively, for the various determinations.

The material was prepared for cooking by removing the bark and sawing the pieces across the grain into sections five-eighths inch thick, which were then split into chips about three-sixteenths to one-fourth inch by 2 to 6 inches across the grain. The chips were screened to remove sawdust, and each lot was thoroughly mixed so as to be uniform throughout.

APPARATUS.

The semicommercial cooks were made in a vertical, stationary digester [1] consisting of a cast-steel cylindrical shell with top and bottom cones, with a capacity of about 62 gallons. The digester was fitted at the top with a "relief" or vent pipe, a pressure gauge, and a thermometer; and at the side with a gauge glass for noting the height of the liquor. The bottom was arranged for "blowing" the contents after cooking. Heat was furnished partly by passing steam directly into the digester at the bottom and partly by two steam coils placed inside the bottom cone. The pressure and temperature were regulated by admitting either more or less steam into the digester and by relieving any excess pressure by means of the top vent.

The autoclave cooks were made in a horizontal rotary autoclave with a capacity of about 2 gallons. This vessel was made of a 6-inch steel pipe with blank flange ends, fitted with trunnions, to one of which was attached a pressure gauge. A screw-joint handhole opening in the side provided for charging. Heat was furnished by Bunsen-burner flames underneath the autoclave, and the pressures were regulated by increasing or decreasing the heat. The autoclave was not relieved during cooking, and no observations of temperatures were made. The cooked pulps were not blown, as in the case of the semicommercial tests, but the cooking vessel was quickly cooled and the contents poured out.

PROCEDURE IN TESTING.

The liquor charges for the sulphate cooks were prepared by mixing caustic soda and sodium sulphide solutions of known composition, as determined by previous analyses, together with water and dry sodium sulphate. The amounts of each constituent were taken in such proportions that when the whole mixture was charged, with the chips,

[1] The apparatus used in the semicommercial cooks is practically the same as that fully illustrated and described in U. S. Department of Agriculture Bulletin No. 80, "Effects of Varying Certain Cooking Conditions in the Productions of Soda Pulp from Aspen," by Henry E. Surface, 1914.

into the digester or autoclave, the amounts of each chemical per pound of chips (bone-dry basis) was in the desired proportion, and the concentration of chemicals in the digester liquor (including the water in the chips) was of the desired degree. For soda cooks the procedure was similar, except that caustic soda was the only chemical to be taken into consideration. The general procedure in conducting the tests was as follows:

The chips to be used for a cook were sampled and weighed. By means of the sample the amount of moisture in the chips and the equivalent bone-dry weight of the charge were determined. The chips, together with the cooking liquors, were then charged into the autoclave or digester, and the vessel closed. After a cook was completed the crude pulp obtained was washed thoroughly, pressed to remove water, shredded, weighed, and sampled for determining its equivalent bone-dry weight. The pulp was then mixed with water and treated in a Hollander-style beating engine[1] with the roll barely touching the bedplate (light brush) until the soft chips in the pulp had become disintegrated into fibers and the wet fibers had a smooth, slippery feel. The beater roll was then pressed hard down on the bedplate (stiff brush), and the beating operation continued until the pulp was suitable for making wrapping paper, as determined by its "feel." The beaten pulp was then screened through the slots (0.012 inch width) of a diaphragm pulp screen. In all cases the screenings obtained were so small in amount that they were disregarded in the yield calculations. The semicommercial pulps were run over a Pusey and Jones 15-inch Fourdrinier paper machine into rolls of dry paper, while the autoclave pulps were made up into sheets on a small hand mold. The papers thus produced contained the experimental pulps alone, without the addition of any other materials.

DETERMINATION OF YIELDS AND PROPERTIES.

The yield of pulp (bone-dry basis) is usually expressed as a percentage of the bone-dry weight of the chip charge, both weights being determined as explained above. When yields per cord are given they are based on a "solid cord" containing 100 cubic feet of clear wood (green volume) having a bone-dry weight of 35.5 pounds per cubic foot;[2] or 3,550 pounds per cord.

The strengths of the papers from the semicommercial pulps were determined by means of a Mullen paper tester, five "pop tests" being made on double thicknesses of each paper. The value is expressed as a "strength ratio," which is the average of the five test values in pounds per square inch divided by the average sheet thicknesses

[1] A 25-pound Emerson beater was used for the semicommercial tests and a 1-pound Noble and Wood beater for the autoclave tests. Both makes were equipped with steel fly bars and steel bedplate bars.

[2] This was the average bone-dry weight of the two butt logs of long leaf pine from Louisiana, the material used in the tests for which yields per cord are given.

in ten-thousandths of an inch, and also as a "strength factor," which is the average of the five pop tests divided by the weight per ream of 500 sheets of paper, each measuring 24 by 36 inches. The relative resistance of the papers to wear was determined by crumpling the sheets in the hand, and all other properties mentioned, except strength, were determined by feel or by observation without the aid of instruments.

DEFINITIONS OF TERMS USED.

While the significance of most of the terms used in recording the test data (Tables 1 to 10, inclusive) is either self-evident or sufficiently clear in view of the previous discussion, there are several which may require explanation.

Water in chips.—The amount of moisture is expressed in percentage of water, based on the calculated bone-dry weight of the chips.

All sodium compounds as Na_2O.—This is the sum of the sodium oxide (Na_2O) equivalents of the amounts of the several constituents entering into the chemical charge. "Total Na_2O" has an analagous significance in the soda process.

Sulphidity.—The sulphidity of the liquor charge is the percentage ratio of the Na_2O equivalent of the amount of sodium sulphide (Na_2S) used to the amount of all sodium compounds present expressed as Na_2O.

Causticity.—This has a similar significance with respect to the amount of caustic soda (NaOH) used.

Initial volume of digester liquors.—The digester liquors include the water in the liquor charge, together with the water in the chips and the water condensed from the steam passed into the digester during cooking. This condensation, of course, does not enter into the calculation of the initial volume.

Apparent condensation.—The apparent condensation is the difference between the calculated initial volume of the digester liquors and the observed volume, as read from a water gauge, at the end of the cook. It roughly represents the amount of steam condensing in the digester during cooking, but does not take into account the volume of the pulp and the differences in temperature of the initial and final liquors, nor the steam and liquid lost during relief.

SEMICOMMERCIAL TESTS.

SULPHATE PROCESS.

The object of the semicommercial sulphate cooks was to secure the best quality of pulp with the highest possible yield. The severity of cooking employed depends largely upon the use for which the pulps are intended. If bleaching or easy bleaching pulps, such as are used in book and other white papers, are desired, more severe cooking

treatments are necessary than if the pulps are to be used in natural-color wrapping papers. The present experiments apply more especially to the latter, for which the important properties are strength, toughness, and resistance to wear. The terms mild, medium, and severe cooking, and undercooked, well-cooked, and overcooked pulps used in the following discussion are significant only with respect to the object of the tests.

MILD COOKING TREATMENTS.

The less severe the cooking of a wood the larger will be the yield of crude pulp. However, there is a point at which the pulp will begin to lose its valuable properties for making wrapping papers. For cook 71 the digesting conditions were outlined to give a much undercooked pulp (see Table 1), but the treatment given the wood was even less severe than is indicated by the recorded data, since a portion of the digester liquor was lost through leakage soon after the cook had been started. The crude unbeaten pulp from this cook was full of soft chips, which, while hard enough to resist the action of a stream of water under pressure, could easily be picked apart with the fingers. The paper made from the beaten pulp had a strength factor of 0.50, was moderately tough, and had fair wearing properties. As a wrapping paper it would be considered of medium grade. The yield, 61.2 per cent, or 2,172 pounds per solid cord, was very high, considering the quality of pulp obtained. Pulps produced under less severe cooking conditions had higher yields (see autoclave tests, pp. 14–24), but the quality was not so good, as evidenced by brittleness, lack of strength, and poor wearing properties.

TABLE 1.—*Record of semicommercial tests using the sulphate process.*

Cook No.	Weight of chips charged (bone-dry basis.)	Water in chips.	Liquor charge.								Initial volume of digester liquors per pound of chips (bone-dry basis).
			Initial concentrations.						Causticity.	Sulphidity.	
			NaOH.	Na_2CO_3.	Na_2S.	SO_2 compounds as Na_2SO_3.	Na_2SO_4.	All sodium compounds as Na_2O.			
	Pounds.	*Per ct.*	*Grams per liter.*	*Grams per liter.*	*Grams per liter.*	*Grams per liter.*	*Grams per liter*	*Grams per liter.*	*Per ct.*	*Per ct.*	*Gallons.*
[1] 71	38.62	34.6	26.5	1.4	13.2	1.8	13.2	38.5	53.3	27.3	0.679
77	38.61	34.7	44.6	2.7	22.3	2.9	22.1	64.9	53.2	27.3	.538
81	23.97	22.7	60.4	3.2	30.0	4.0	30.0	87.6	53.4	27.2	.300
85	23.97	22.7	36.0	1.9	18.0	2.4	18.0	52.3	53.3	27.3	.500
92	23.97	22.6	48.0	2.4	30.0	4.0	30.0	77.5	48.0	30.8	.300
98	23.97	22.6	28.8	1.4	14.4	1.9	14.4	41.8	53.3	27.4	.500
113	25.38	18.2	34.2	1.9	17.1	2.2	18.5	50.4	52.6	27.0	.700
138	25.38	18.2	59.9	3.0	30.0	3.9	30.0	87.0	53.4	27.4	.400
141	25.38	18.2	60.0	3.3	15.0	2.1	30.0	74.4	62.4	16.0	.400
146	25.38	18.2	26.5	1.4	13.2	1.8	13.2	38.5	53.3	27.3	.680
147	26.67	12.5	26.5	1.2	13.2	1.8	13.2	38.4	53.4	27.4	.680
148	26.67	12.5	26.5	1.2	13.2	1.8	13.2	38.4	53.4	27.4	.680

[1] A portion of the digester liquor was lost, due to leaks during the early stages of cooking.

TABLE 1.—*Record of semicommercial tests using the sulphate process*—Continued.

Cook No.	Chemicals charged per 100 pounds of chips (bone-dry basis).						Duration of cooking.			Maximum cooking temperature.	
	NaOH.	Na_2CO_3.	Na_2S.	SO_2 compounds as Na_2SO_3.	Na_2SO_4.	All sodium compounds as Na_2O.	Total.	At zero gauge pressure.	At maximum gauge pressure.		
	Pounds.	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Hours.*	*Hours.*	*Hours.*	°*F.*	°*C.*
[1] 71	15.0	0.8	7.5	1.0	7.5	21.8	3.0	0.1	2.8	331	166
77	20.0	1.2	10.0	1.3	9.9	29.1	3.0	.2	2.3	331	166
81	15.1	.8	7.5	1.0	7.5	21.9	3.0	.1	2.5	331	166
85	15.0	.8	7.5	1.0	7.5	21.8	3.0	.1	2.5	331	166
92	12.0	.6	7.5	1.0	7.5	19.4	3.0	.25	2.5	331	166
98	12.0	.6	6.0	.8	6.0	17.4	5.0	.1	4.3	331	166
113	20.0	1.1	10.0	1.3	10.8	29.4	3.0	.25	1.0	331	166
138	20.0	1.0	10.0	1.3	10.0	29.0	3.0	.1	2.8	331	166
141	20.0	1.1	5.0	.7	10.0	24.8	3.0	.1	2.5	331	166
146	15.0	.8	7.5	1.0	7.5	21.8	3.0	.2	2.3	338	170
147	15.0	.7	7.5	1.0	7.5	21.8	3.5	.3	2.8	338	170
148	15.0	.7	7.5	1.0	7.5	21.8	3.5	.2	3.0	338	170

Cook No.	Digester pressures per square inch.		Steam pressure per square inch at digester inlet.	Apparent condensation per pound of chips (bone-dry basis).	Yield of crude pulp (bone-dry basis).		Duration of beater treatment.			Strength ratio of paper.	Strength factor of paper.	Ream weight of papers tested.
	Maximum gauge.	Blowing.					Total.	At light brush.	At stiff brush.			
	Pounds.	*Pounds.*	*Pounds.*	*Gallons.*	*Per*	*Lbs. per solid cord.*	*Hours.*	*Hours.*	*Hours.*			*Pounds.*
[1] 71	90	40	105	[1] 0.20	61.2	2,172	3.5	1.5	2.0	0.60	0.50	76
77	90	40	105	.50	45.3	1,609	3.5	2.0	1.5	1.15	.91	31
81	90	50	103	.58	47.9	1,700	5.0	2.5	2.5	1.08	.93	44
85	90	50	108	.....	52.0	1,846	7.0	3.0	4.0	.91	.87	38
92	90	40	110	.49	48.8	1,733	6.5	2.5	4.0	.86	.70	28
98	90	40	108	.50	51.8	1,839	4.5	1.0	3.5	.60	.56	28
113	90	40	100–95	.32	48.6	1,725	6.0	2.0	4.0	.70	.59	37
138	90	40	110	.....	46.1	1,637	.....	.....	.....	.....	.....	.....
141	90	40	108	.....	44.2	1,569	8.5	4.5	4.0	1.02	.86	36
146	100	40	105	.....	54.9	1,949	9.0	4.0	5.0	.72	.68	45
147	100	40	115	.41	49.1	1,743	6.5	4.0	2.5	.92	.71	37
148	100	40	115	.63	48.4	1,718	8.5	4.0	4.5	1.02	.77	33

(P. L.—138, S. L.—176.)

[1] A portion of the digester liquor was lost, due to leaks during the early stages of cooking.

SEVERE COOKING TREATMENTS.

The effect of more severe cooking treatments, produced mainly by greater initial concentrations and amounts of active cooking chemicals, was evidenced by the thoroughly cooked or overcooked pulps from cooks 77 and 141 (Table 1). The crude pulps were not only free from chips and shives, but also seemed to be soft and fluffy. The papers made from the beaten pulps, however, were of very superior quality with regard to resistance to wear, toughness, and strength, the strength factors being 0.91 and 0.86 for cooks 77 and 141, respectively. Both pulps became slightly hydrated during the beater treatments, which produced a parchmentizing effect and increased the strength and toughness. Either of the papers could be

rubbed or crumpled for a long time without becoming fuzzy, tearing, or showing signs of wear at the place of friction. The papers had also a soft, smooth, greasy, leather-like feel, and were light brown in color, like the imported kraft papers. The yields were rather low for sulphate kraft pulps. For cook 77 the yield was 45.3 per cent, or 1,609 pounds per solid cord, and for cook 141, 44.2 per cent, or 1,569 pounds per solid cord. Under still more severe cooking treatments longleaf pine pulps become very soft and gradually lose their strength and wearing properties. (See autoclave tests, p. 14–24.)

MEDIUM COOKING TREATMENTS.

The above-mentioned cooks show approximately the higher and lower limits of yield in the production of pulps and papers of good quality. However, the better quality of wrapping papers resulted from pulps having the lower yields, and in attempting to secure this better quality, but with higher yields than were obtained for cooks 77 and 141, cooks 85, 98, and 146 were made. For cook 85 the amounts of chemicals and the initial concentrations were decreased from the corresponding conditions for cook 77, while the duration of cooking and the pressure remained practically the same; for cook 98 a further decrease was made in the amounts of chemicals and in the concentrations, but the duration of cooking was increased; for cook 146 the amounts of chemicals and the duration were practically the same as for cook 85, but the concentrations were decreased while the pressure was increased. The cooking conditions, given in full in Table 1, are briefly summarized in Table 2. The resultant papers were in each case of good quality, being tough and resistant to wear, but they were in general not so strong as those from pulps produced under more severe cooking treatments. The strength factors for cooks 85, 98, and 146 were 0.87, 0.56, and 0.68, respectively. There is little doubt, however, that these values could be increased considerably by employing beating and other refining treatments better adapted for these particular pulps than the treatments given them. The yields obtained were quite high, cook 85 yielding 52 per cent, or 1,846 pounds per solid cord; cook 98, 51.8 per cent, or 1,839 pounds per solid cord; and cook 146, 54.9 per cent, or 1,949 pounds per solid cord.

TABLE 2.—*Condensed summary of cooking conditions for cooks 77, 85, 98, and 146.*

Cook No.	Liquor charge, initial concentrations.		Chemicals per 100 pounds of chips (bone-dry basis).		Duration of cooking.		Maximum gauge pressure per square inch.
	NaOH.	Na_2S.	NaOH.	Na_2S.	Total.	At maximum gauge pressure.	
	Grams per liter.	*Grams per liter.*	*Pounds.*	*Pounds.*	*Hours.*	*Hours.*	*Pounds.*
77	44.6	22.3	20.0	10.0	3.0	2.3	90
85	36.0	18.0	15.0	7.5	3.0	2.5	90
98	28.8	14.4	12.0	6.0	5.0	4.3	90
146	26.5	13.2	15.0	7.5	3.0	2.3	100

All things considered, cooks 147 and 148, which may also be classed with those of medium severity, gave the best results. These two cooks were made under almost duplicate cooking conditions, approximately as follows: Caustic soda and sodium sulphide charged per 100 pounds of chips, 15 and 7.5 pounds, respectively; initial concentration of caustic soda in digester liquor, 26.5 grams per liter; initial volume of digester liquor per pound of chips, 0.68 gallon; total duration of cooking, 3.5 hours, of which 2.8 hours for cook 147 and 3.0 hours for cook 148 were at a maximum gauge pressure of 100 pounds per square inch.

The crude pulps were slightly raw and contained some soft chips, which, however, broke up in the beater. The pulp from cook 148 was hydrated during the beating treatment to such an extent that the paper made from it had a parchment-like appearance, the individual fibers being scarcely distinguishable from each other. This paper had good wearing properties and was very tough, with a strength factor of 0.77. The pulp from cook 147 was not subjected to so long a beating treatment, and the resulting paper was not parchmentized to the same extent as that from cook 148. It had a strength factor of 0.71, however, was very tough, and showed good wearing properties. The yield from cook 148 was 48.4 per cent, or 1,718 pounds per solid cord, and from cook 147, 49.1 per cent, or 1,743 pounds per solid cord.

EFFECTS OF BEATING.

The mechanical treatment given a kraft pulp has as important an influence on the properties of the resulting paper as the cooking treatment itself. A crude pulp which appears to be of little value can be made into strong high-grade paper if the proper beater treatment is employed, while the best pulps can easily be ruined by improper beating. The use of kollergangs or edge runners preliminary to actual beating, or of stone rolls and bedplates in the beaters, and the determination by successive tests of the refining and beating treatments best adapted for a particular pulp undoubtedly would have resulted in papers of much better quality than those obtained. Nevertheless, many of the experimental papers were equal or superior to commercial kraft papers.

The effect of different beater treatments was shown by a single series of tests on some of the crude pulp from cook 71 (Table 1). Separate portions of the pulp were treated in the 1-pound beater for periods of 0.5, 1, 2, 3, and 4 hours with the roll at light brush. The papers resulting from treatments of 2 hours or less were soft and weak, and had poor wearing properties, but for the longer periods the papers were firm and tough, with good wearing properties. Under the 4-hour treatment the fibers became hydrated, and a parchment-like paper resulted. The fibers of longleaf pine when reduced by the sulphate process seem to take up water and to become hydrated very

quickly. For all of the semicommercial tests previously mentioned (Table 1) this effect, indicated by the smooth, greasy feel of the wet paper stock, was obtained with from 2 to 4 hours' beater treatment.

WOOD REQUIRED FOR 1 TON OF PULP.

It has been shown that sulphate kraft pulps of fairly good strength and toughness can be obtained from longleaf pine with yields (bone-dry basis) as high as 61 per cent, or 2,170 pounds per solid cord [1] in case of wood as heavy as that tested. For the production of the best grades of wrapping papers, which equal or excel in quality the imported sulphate kraft papers, the yield of pulp would be approximately 51 per cent, or 1,800 pounds (bone-dry) per solid cord. This is equal to a ton (2,000 pounds) of nominally air-dry pulp.[2] However, it should be remembered that for wood either lighter or heavier than that on which this calculation is based the amount required per ton of pulp would be correspondingly greater or less, unless the differences in weight were due to resin alone.[3]

COMPARISON OF THE SODA AND SULPHATE PROCESSES.

Table 3 contains the record of the semicommercial soda tests. The best results in both yield and quality were obtained in the case of cook 152. This cook employed 20 pounds of caustic soda per 100 pounds of wood at an initial concentration of 79.7 grams per liter and 5 hours' cooking at 110 pounds gauge pressure, the total duration being 6 hours. The resulting paper was very strong (strength factor 0.90) and the feel and wearing properties were also exceptionally good for a soda pulp. The yield was 48 per cent, or 1,704 pounds per solid cord.

TABLE 3.—*Record of semicommercial tests using the soda process.*

Cook No.	Weight of chips charged (bone-dry basis).	Water in chips.	Liquor charge.				Inital volume of digester liquor per pound of chips (bone-dry basis).	Chemicals charged per 100 pounds of chips (bone-dry basis).		
			Initial concentrations.			Causticity.				
			NaOH.	Na_2CO_3.	Total. Na_2O.			NaOH.	Na_2CO_3.	Total. Na_2O.
	Pounds.	*Per cent.*	*Grams per liter.*	*Grams per liter.*	*Grams per liter.*	*Per cent.*	*Gallons.*	*Pounds.*	*Pounds.*	*Pounds.*
102	23.97	22.6	84.0	3.4	67.1	97.0	0.538	37.7	1.5	30.1
136[1]	25.37	18.3	59.9	2.3	47.8	97.2	.400	20.0	7.7	16.0
144	25.38	18.2	90.0	3.4	71.7	97.2	.400	30.0	1.1	23.9
149	25.89	12.0	90.2	3.2	71.8	97.4	.332	25.0	.9	19.9
150	25.89	12.0	90.2	3.2	71.8	97.4	.332	25.0	.9	19.9
151	25.89	12.0	90.2	3.2	71.8	97.4	.332	25.0	.9	19.9
152	25.89	12.0	79.7	1.8	62.9	98.3	.301	20.0	.5	15.8
176–1[2]	41.89	14.6	90.0	2.7	71.3	97.8	.333	25.0	.8	19.8
176–2[2]	41.89	14.6	90.0	2.42	71.2	98.0	.266	20.0	.5	15.8
176–3[2]	41.89	14.6	90.0	2.42	71.1	98.0	.267	20.0	.5	15.8

[1] Weighing 3,550 pounds; see p. 6.

[2] Standard moisture content of 10 per cent or 100 pounds air-dry weight equals 90 pounds bone-dry weight.

[3] The average specific gravity (oven-dry weight, green volume) of all of the longleaf pine from Louisiana in the shipment from which the two test logs were taken, including bolts cut higher up in the trunks of the same trees and material from several additional trees, was 0.528. (See Forest Service Circular 213, Mechanical Properties of Woods Grown in the United States, 1913, Table 1.) This is equal to a weight per cubic foot of 33 pounds in comparison with the 35.5 pounds obtained for the two butt logs.

TABLE 3.—*Record of semicommercial tests using the soda process*—Continued.

Cook No.	Duration of cooking.			Maximum cooking temperature.		Digester pressures per square inch.		Steam pressure per square inch at digester inlet.	Apparent condensation per pound of chips (bone-dry basis).
	Total.	At zero gauge pressure.	At maximum gauge pressure.			Maximum gauge.	Blowing.		
	Hours.	*Hours.*	*Hours.*	*° F.*	*° C.*	*Pounds.*	*Pounds.*	*Pounds.*	*Gallons.*
102	3.0	0.2	2.5	331	166	90	40	115	0.55
136[1]	6.0	.2	5.3	331	166	90	45	115	.55
144	6.0	.2	5.5	338	170	100	40	110	.80
149	6.0	.2	5.0	338	170	100	40	122	.....
150	9.3	.3	8.3	307	153	60	40	120	.....
151	4.5	.2	3.8	361	183	140	40	142	1.02
152	6.0	.2	5.0	345	174	110	40	125	.55
176-1[2]	7.0	.5	6.0	338	170	100	.....	110	.94
176-2[2]	7.0	.3	6.0	338	170	100	.....	115	.76
176-3[2]	8.0	.3	7.0	338	170	100	.....	110	.91

Cook No.	Causticity of black liquor at end of cook.	Efficiency in use of NaOH.	Yield of crude pulp (bone-dry basis).		Duration of beater treatment.			Strength ratio of paper.	Strength factor of paper.	Ream weight of papers tested.
					Total.	At light brush.	At stiff brush.			
	Per cent.	*Per cent.*	*Per cent.*	*Lbs. per solid cord.*	*Hours.*	*Hours.*	*Hours.*			*Pounds.*
102	.....	.....	47.2	1,676	4.0	2	2.0	0.43	0.41	42
136[1]	.....	.....	50.9	1,808	6.5	2	4.5	.55	.54	60
144	.....	.....	39.8	1,413	6.0	6	0.0	1.04	.84	48
149	22.7	76.7	44.1	1,566	6.5	2	4.5	1.04	.84	37
150	41.6	57.3	52.0	1,846	9.0	5	4.0	.53	.56	43
151	17.6	81.9	37.6	1,335	6.0	6	0.0	.91	.80	46
152	11.2	88.6	48.0	1,704	8.5	4	4.5	1.05	.90	37
176-1[2]	.....	.....	48.6	.....	.....	.....	.....	.....	.....	.....
176-2[2]	.....	.....	51.1	.....	.....	.....	.....	.....	.....	.....
176-3[2]	.....	.....	50.4	.....	.....	.....	.....	.....	.....	.....

(P. L.—138, S. L.—176; P. L.—164-1.)

[1] Five pounds of sodium chloride (table salt) per 100 pounds of chips were used in addition to the chemicals indicated. However, it will be shown later that sodium chloride has little or no effect. (See p. 19.)

[2] Shipment L-3*a* from Mississippi was used as the test material. Data for these three cooks have been published previously in Forest Service unnumbered bulletin, "Paper Pulps from Various Forest Woods," by Henry E. Surface, 1912. Specimens of natural color and bleached pulps accompanied the data.

Cook 150 afforded a yield of 52 per cent, or 1,846 pounds per solid cord, but the quality was not so good as in the case of cook 152, the paper being quite weak (strength factor 0.56) with a correspondingly low resistance to wear. The papers resulting from cooks 144, 149, and 151 were all of very good quality, having high strength ratios and good wearing properties, but the yields were considerably lower than for cook 152.

Soda pulps from longleaf pine tend to be soft and fluffy, even when slightly undercooked, or chippy. Proper beater treatments will remedy this to some extent, but the pulp does not become so well hydrated nor attain the same smooth, greasy feel during beating as the sulphate pulps, and the resultant papers do not show the parchmentized effect so characteristic of the sulphate papers. On the paper machine soda stock runs "free," while sulphate stock runs "slow," provided, of course, both kinds of stock are handled similarly in the beater.

The soda papers were inferior to the sulphate papers in resistance to wear; the latter could be rubbed and crumpled for a long time without showing signs of wear, while the former had a tendency to become fuzzy and tear under similar treatment. Even those sulphate pulps at very high yields had wearing qualities equal to the best soda pulps. There is little doubt that higher yields of good kraft pulp can be obtained with the sulphate process than with the soda process. Sulphate pulps of fairly good quality can be obtained with yields as high as 61 per cent, while the limit for soda pulps is approximately 50 per cent. With higher yields the soda pulps lose strength and toughness and become brittle. A sulphate pulp with a 60 per cent yield can be made into a medium grade of kraft wrapping paper, while a soda pulp having the same yield will produce only a very inferior grade. Considering bursting strength alone, equally strong papers can be made by either process.

The main advantage of the sulphate process over the soda process is that in the former the pulp can be very much undercooked and still produce a fair quality of paper, while a soda pulp must be comparatively well cooked before a good paper can be made from it. Moreover, the best sulphate kraft pulps were obtained with a total duration of cooking of only 3.5 hours, while in the soda tests 6 hours were required to secure the best results.

AUTOCLAVE TESTS.

The autoclave tests, which, as previously explained, preceded the semicommercial tests, were made to determine the effects of varying the cooking conditions in the production of sulphate pulp. The cooking conditions investigated were:

(1) Amounts of the various cooking chemical employed.
(2) Cooking pressures or temperatures.
(3) Durations of cooking.
(4) Initial concentrations of chemicals in the digester liquors.

Aside from the chemicals normally present in sulphate cooking liquors—that is, caustic soda, sodium sulphide, sodium sulphate, and sodium carbonate, the effects of sodium chloride and sulphur in conjunction with caustic soda were studied. The tests, Tables 4 to 10, inclusive, were made in series, in any of which all cooking conditions except the one under observation were held as nearly constant as possible.

The amounts of sodium carbonate and of SO_2 compounds expressed as Na_2SO_3 in the cooking liquors were in general small and no mention of them is made in the tabulated data. The amounts of sodium sulphate present are indicated only relatively, except in Tables 6 and 10.

EFFECTS OF VARYING AMOUNTS OF CAUSTIC SODA.

The effect of varying amounts of caustic soda on the yield of crude pulp is shown in Table 4. Two series of tests were made, differing in the amounts of sodium sulphate and sodium sulphide employed. In the first series increasing the amounts of caustic soda from 15 to 90 pounds per 100 pounds of wood resulted in a decrease in the yield of from 52 to 27.7 per cent. This decrease, however, was not directly proportional to the increase of caustic soda used, as values of this chemical between 30 and 50 pounds had little effect in varying the yield. For higher and lower values the effect was quite pronounced. In the second series a larger amount of sodium sulphide was used, and consequently the yields were lower for corresponding amounts of caustic soda, but variations in the amounts of this chemical produced similar effects.

TABLE 4.—*Effect of varying amounts of caustic soda (NaOH) on the yield of pulp.*

Weight of chips charged (bone-dry basis)	pounds	0.986 to	1.007
Water in chips	per cent	10.2 to	12.6
Initial volume of digester liquors per pound of chips (bone-dry basis)	gallons	0.650 to	0.690
Duration of cooking, total	hours		3.0
Duration of cooking at zero gauge pressure	do		0.1
Duration of cooking at maximum gauge pressure	do		2.3
Maximum gauge pressure per square inch	pounds		90
Total duration of beater treatment (at light brush only)	hours		0 or 1

FIRST SERIES.

Cook No.	Liquor charge. Initial concentrations. NaOH.	Na_2S.[1]	All sodium compounds, as Na_2O.	Causticity.	Sulphidity.	Chemicals charged per 100 pounds of chips (bone-dry basis). NaOH.	Na_2S.[1]	All sodium compounds, as Na_2O.	Yield of crude pulp (bone-dry basis).
	Grams per liter.	*Grams per liter.*	*Grams per liter.*	*Per cent.*	*Per cent.*	*Pounds.*	*Pounds.*	*Pounds.*	*Per cent.*
31	26.3	13.1	38.1	53.6	27.5	15.0	7.5	21.7	52.0
55	52.1	13.0	57.9	69.7	17.9	30.0	7.5	33.4	42.9
56	69.6	13.0	72.0	74.9	14.4	40.0	7.5	41.4	39.6
57	87.0	13.0	85.6	78.7	12.1	50.0	7.5	49.2	42.1
58	104.4	13.0	99.7	81.1	10.4	60.0	7.5	57.3	40.0
59	121.8	13.0	113.6	83.1	9.1	70.0	7.5	65.3	33.3
60	156.6	13.0	139.3	87.1	7.4	90.0	7.5	80.1	27.7

SECOND SERIES.

29	35.4	44.4	84.2	32.6	42.0	19.9	25.0	47.4	42.3
28	55.3	45.9	101.9	42.1	35.8	30.0	24.9	55.2	38.2
33	52.7	43.9	99.5	41.0	35.1	30.0	25.0	56.6	37.0
54	60.8	43.3	104.5	45.1	32.9	35.0	24.9	60.1	39.9
32	70.1	43.9	113.0	48.5	30.9	39.9	25.0	64.4	34.8
27	73.8	45.9	116.7	49.0	31.3	40.0	24.9	63.3	35.0
53	76.6	43.3	117.0	50.8	29.4	44.1	24.9	67.3	38.6
43	88.2	44.3	128.3	53.2	27.4	49.8	25.0	72.5	36.8
26	88.8	44.4	126.8	54.3	27.9	50.0	25.0	71.4	37.6
49	104.2	43.4	139.2	58.0	24.8	60.0	25.0	80.2	31.8
52	121.6	43.2	152.4	61.9	22.6	70.0	24.9	87.8	31.8
51	139.0	43.5	166.6	64.6	20.7	80.0	25.0	95.8	28.1

(P. L.—138, S. L.—3b.)

[1] With a few minor exceptions, the same values apply to the Na_2SO_4.

The best quality of pulp was obtained with cook 31, using 15 pounds of caustic soda per 100 pounds of wood. This resulted in a slightly undercooked product, which came from the autoclave in the

form of soft chips. The chips did not break up during the washing operation, but were readily pulped by beater treatment. The pulp was strong, tough, and resistant to wear. When larger amounts of caustic soda were employed the pulp tended to be soft, fuzzy, and less strong, while for smaller amounts it was harsh and brittle. (See cooks 39 and 40, Table 6.) In the second series of tests (Table 4) the conditions were such that all of the pulps were overcooked if considered for kraft papers.

The higher the amount of caustic soda employed, the lighter in color was the pulp. The extremes for the first series of tests were brown in the case of cook 31 and light gray in the case of cook 60. For the second series of tests the color change was less noticeable.

EFFECTS OF VARYING AMOUNTS OF SODIUM SULPHIDE.

The effects of varying the amount of sodium sulphide were shown by three series of tests employing different amounts of caustic soda and of sodium sulphate. The cooking conditions and resultant yields are given in Table 5.

TABLE 5.—*Effect of varying amounts of sodium sulphide (Na_2S) on the yield of pulp.*

Weight of chips charged (bone-dry basis)	pounds..	0.996 to 1.043
Water in chips	per cent..	11.0 to 16.0
Initial volume of digester liquors per pound of chips (bone-dry basis)	gallons..	0.662 to 0.683
Duration of cooking, total	hours..	3.0
Duration of cooking at zero gauge pressure	do....	0.1
Duration of cooking at maximum gauge pressure	do....	2.0 to 2.5
Maximum gauge pressure per square inch	pounds..	90
Total duration of beater treatment (at light brush only)	hours..	0, 1, or 2

FIRST SERIES.[1]

Cook No.	Liquor charge. Initial concentrations. NaOH.	Na_2S.[2]	All sodium compounds, as Na_2O.	Causticity.	Sulphidity.	Chemicals charged per 100 pounds of chips (bone-dry basis). NaOH.	Na_2S.[2]	All sodium compounds, as Na_2O.	Yield of crude pulp (bone-dry basis).
	Grams per liter.	*Grams per liter.*	*Grams per liter.*	*Per cent.*	*Per cent.*	*Pounds.*	*Pounds.*	*Pounds.*	*Per cent.*
31	26.3	13.1	38.1	53.6	27.5	15.0	7.5	21.7	52.0
34	26.3	26.3	49.6	41.1	42.2	15.0	15.0	28.3	47.4
35	26.3	43.9	62.4	32.7	56.0	15.0	25.0	35.5	44.5
36	26.3	70.2	88.0	23.2	63.5	15.0	40.0	50.0	39.9
37	26.5	88.3	103.8	19.7	67.5	15.0	50.0	58.9	40.3
SECOND SERIES.[1]									
133	27.2	1.8	31.1	67.7	4.6	15.0	1.0	17.2	68.9
134	27.2	5.4	34.4	61.3	12.6	15.0	3.0	19.0	67.6
139	27.0	9.0	37.3	56.1	19.2	15.0	5.0	20.7	60.1
THIRD SERIES.[1]									
129	35.3	3.7	39.4	69.4	7.4	20.0	2.1	22.3	64.3
130	35.9	7.2	43.0	64.8	13.3	20.0	4.0	23.9	53.7
131	36.0	10.8	46.3	60.2	18.5	20.0	6.0	25.7	49.7
132	36.0	14.4	49.6	56.2	23.1	20.0	8.0	27.6	47.7

(P. L—138.)

[1] The Mississippi wood (shipment L-3*b*) was used for the first series and the Louisiana wood (shipment L-176) for the second and third series.

[2] With a few minor exceptions, the Na_2SO_4 amounted to one-half of these values for the first and third series and to two-thirds of these values for the second series.

In the first series of tests, with an increase in the amount of sodium sulphide from 7.5 to 50 pounds per 100 pounds of wood, the yield decreased from 52 to 40.3 per cent. For amounts of 25 pounds or less the pulps were of good quality, being strong with good wearing properties, but for larger amounts the pulps became soft and fuzzy and evidently were overcooked.

In the second series, increasing the amount of sodium sulphide from 1 to 5 pounds per 100 pounds of wood resulted in a decrease in the yields from 68.9 to 60.1 per cent. The largest amount (5 pounds) afforded the best pulp, considering strength and wearing properties; the other pulps were much undercooked and quite brittle.

The third series of tests, using a larger amount of caustic soda (20 pounds), showed the effect of increasing the amount of sodium sulphide from 2.1 to 8 pounds per 100 pounds of wood. Under these conditions, the yield was decreased from 64.3 to 47.7 per cent. The pulp obtained when using 2.1 pounds of sodium sulphide was slightly undercooked and somewhat brittle. The other pulps had fair strength and wearing properties and could be used for making a medium grade of wrapping paper.

As the amount of sodium sulphide was increased, the disagreeable odor arising from the cooking was more noticeable, being much more offensive for cook 37 (50 pounds Na_2S per 100 pounds of wood) than for cook 31 (7.5 pounds Na_2S). Increasing the amount of sodium sulphide resulted in lighter-colored pulps, that from cook 37 being considerably lighter in color than from cook 31.

Sodium sulphide is not so severe in its action on wood as caustic soda. A cook of 8 hours' duration was made with sodium sulphide only, using 40 pounds per 100 pounds of wood and a maximum cooking pressure of 100 pounds per square inch. A yield of 41 per cent was obtained, while a similar cook using caustic soda alone in the proportion of 20 pounds per 100 pounds of wood had a yield of 44.3 per cent. This indicates that caustic soda is almost twice as effective as sodium sulphide in reducing the wood to pulp. The color of the pulp produced when using caustic soda alone was lighter than when using sodium sulphide alone.

EFFECTS OF SODIUM CARBONATE.

Sodium carbonate occurs in the commercial sulphate liquors due to incomplete causticization. That it is of no assistance in reducing longleaf pine was shown by a cook made with 40 pounds of this chemical, 10 pounds of caustic soda, and 5 pounds of sodium sulphide per 100 pounds of wood. The duration of cooking was 7 hours and

the maximum gauge pressure was 100 pounds per square inch. The product came from the autoclave in the form of hard, black chips which were quite "raw" on the inside; the yield was not determined. In comparison with this result, cook 40 (Table 6), using, per 100 pounds of wood, 10 pounds of caustic soda, 5 pounds of sodium sulphide, and 5 pounds of sodium sulphate (the latter being of no assistance in cooking), also afforded a product in the chip form. These chips, however, were soft, and could easily be picked apart with the fingers. Of the 3 hours' total duration for this cook, 2.3 hours were at a maximum pressure of 90 pounds. The yield was 65.7 per cent. While it is hardly safe to base a general conclusion upon a single trial, this test indicates that sodium carbonate, at least when present in considerable quantity, retards or diminishes the effects of the caustic soda and sodium sulphide.

EFFECTS OF SODIUM SULPHATE.

Sodium sulphate is present in the commercial cooking liquors, due to incomplete reduction of the sulphate to sulphide during the smelting operations. Like sodium carbonate, it is of practically no assistance in cooking. A cook of 3 hours' duration and 90 pounds maximum gauge pressure was made, using sodium sulphate in the proportion of 50 pounds per 100 pounds of wood, which yielded 86.3 per cent, while another cook of the same duration and pressure but without any chemicals whatever (that is, using pure water alone) had a yield of 89.1 per cent. Allowing for experimental errors, there was little difference between the results of these two cooks, and in neither case could the product be beaten into pulp.

A cook was also made, using 40 pounds of sodium sulphate, 10 pounds of caustic soda, 5 pounds of sodium carbonate, and 5 pounds of sodium sulphide per 100 pounds of wood; the duration was eight hours and the maximum gauge pressure was 100 pounds per square inch. Only hard black chips were obtained, of no value whatever for pulp. As in the case of the sodium carbonate, there is an indication that sodium sulphate retards the action of the other chemicals. To prove this further tests are necessary.

EFFECTS OF VARYING ALL CHEMICALS IN SAME PROPORTION.

A series of tests was made varying the amounts of all sodium compounds present in sulphate cooking liquors. The several constituents were kept constant in regard to each other in the proportion of 50 parts caustic soda, 25 parts sodium sulphide, and 25 parts sodium sulphate. For convenience the amounts of the different chemicals have been computed to a common basis, and the combined values are expressed as Na_2O (sodium oxide).

The yields shown in Table 6 varied from 65.7 per cent for 14.5 pounds of total Na_2O per 100 pounds of wood to 36.8 per cent for 72.5 pounds of total Na_2O. The conditions indicated for cook 31 afforded the best results with regard to both yield and quality of pulp produced. With the higher yields the pulps were harsh and had less resistance to wear. Nevertheless, wrapping papers of medium grades could be made from these pulps. The pulp from cook 30 was of good quality, with strength and wearing properties equal to that from cook 31, but the yield was not so high. Cooks 43, 26, and 38 were duplicates of each other, and show the accuracy attained in the yield determinations. The pulps from these three cooks were soft and fluffy, and had poor strength and wearing properties, due to overcooking.

TABLE 6.—*Effect of varying amounts of all sodium compounds on the yield of pulp.*

Weight of chips charged (bone-dry basis)	pounds	0.996 to 1.007
Water in chips	per cent	10.2 to 11.5
Causticity of liquor charge	do	53.2 to 54.3
Sulphidity of liquor charge	do	27.4 to 27.9
Initial volume of digester liquors per pound of chips (bone-dry basis)	gallons	0.675 to 0.683
Duration of cooking, total	hours	3.0
Duration of cooking, at zero gauge pressure	do	0.1
Duration of cooking, at maximum gauge pressure	do	2.0 to 2.3
Maximum gauge pressure per square inch	pounds	90
Total duration of beater treatment (at light brush only)	hours	0, 1, or 2

Cook No.	Liquor charge, initial concentrations.				Chemicals charged per 100 pounds of chips (bone-dry basis).				Yield of crude pulp (bone-dry basis).
	NaOH.	Na_2S.	Na_2SO_4.	All sodium compounds as Na_2O.	NaOH.	Na_2S.	Na_2SO_4.	All sodium pounds as Na_2O.	
	Grams. per liter.	*Grams. per liter.*	*Grams. per liter.*	*Grams. per liter.*	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Per cent.*
40	17.6	8.8	8.8	25.5	10.0	5.0	5.0	14.5	65.7
39	21.2	10.6	10.6	30.7	12.0	6.0	6.0	17.4	60.2
31	26.3	13.1	13.1	38.1	15.0	7.5	7.5	21.7	52.0
30	35.6	17.8	17.8	51.3	20.0	10.0	10.0	28.9	47.0
43	88.2	45.3	45.3	128.3	49.8	25.0	25.6	72.5	36.8
26	88.8	44.4	44.4	126.8	50.0	25.0	25.0	[1] 71.4	37.6
38	88.2	44.1	44.1	127.6	50.0	25.0	25.0	[1] 72.4	36.6

(P. L.—138, S. L.—3*b*.)

[1] The Na_2O values for cooks 26 and 38 differ mainly because of different amounts of Na_2CO_3 which are not separately recorded in the table.

EFFECTS OF SODIUM CHLORIDE.

A few tests were made to determine whether or not the use of sodium chloride in conjunction with caustic soda would result in firmer and less fuzzy pulps, more resistant to wear, than are ordinarily produced with the soda process. If this were possible a process might be developed to produce pulps similar to those obtained with the sulphate process without the disagreeable odors so characteristic of it. Table 7 shows a comparison between cooks made with caustic soda alone and with caustic soda and sodium chloride. It is not probable that sodium

chloride has an effect on the yield, as is evidenced by the data for cooks 128 and 137. Both cooks employed 20 pounds of caustic soda per 100 pounds of wood, but the former used 5 pounds of sodium chloride in addition. The yields from the two cooks were identical. The use of sodium chloride appeared to improve the qualities of the pulps somewhat, but they were much inferior to sulphate pulps at similar yields. The few advantages attending the use of sodium chloride preclude the possibility of this modification of the soda process being of commercial value.

TABLE 7.—*Effect of sodium chloride (NaCl) used in conjunction with caustic soda (NaOH) on the yield of pulp.*

Weight of chips charged (bone-dry basis)	pounds	0.910 to 1.043	
Water in chips	per cent	15.1 to 22.0	
Causticity of liquor charge (disregarding NaCl)	do	96.0 to 97.2	
Duration of cooking at zero gauge pressure	hours	0.1	
Maximum gauge pressure per square inch	pounds	90	
Total duration of beater treatment (at light brush only)	hours	2	

Cook No.	Liquor charge, initial concentrations.		Chemicals charged per 100 pounds of chips (bone-dry basis).		Initial volume of digester liquors per pound of chips (bone-dry basis).	Duration of cooking.		Yield of crude pulp (bone-dry basis).
	NaOH.	NaCl.[1]	NaOH.	NaCl.[1]		Total.	At maximum gauge pressure.	
	Grams per liter.	*Grams per liter.*	*Pounds.*	*Pounds.*	*Gallons.*	*Hours.*	*Hours.*	*Per cent.*
118	44.2	28.6	15.0	10.0	0.420	3.0	2.5	73.9
122	41.1	20.0	20.0	10.0	.600	4.0	3.5	63.5
128	49.2	12.0	20.0	5.0	.500	6.0	5.3	58.9
137	36.3	0	20.0	0	.662	6.0	5.3	58.9
72	35.2	0	20.0	0	.681	3.0	2.3	71.6

(P. L.—138, S. L.—176.)

[1] The values shown represent common table salt and not the pure chemical.

EFFECTS OF SULPHUR.

Cooks using "flowers of sulphur" and caustic soda as the cooking chemicals produced pulps almost identical with those resulting from the sulphate process. The addition of sulphur undoubtedly imparted to the pulps the resistance to wear and strength not obtainable by the soda process alone. These cooks, however, were characterized by the same disagreeable odor as the sulphate cooks, and this modification of the soda process seems to have no particular technical advantage over the sulphate process except in the matter of control of the cooking liquors.

EFFECTS OF VARYING THE PRESSURES OR TEMPERATURES OF COOKING.

In the sulphate process, as in the soda process, the digester pressures represent the pressure of saturated steam, since no other gases are present in sufficient quantity to affect the pressure. This

was determined by actual test. The digester pressures, therefore, correspond to the temperatures of saturated steam; and values of each may be converted into the other by means of standard steam tables.

Table 8 shows the effect on yield of variations of pressure from 40 to 140 pounds per square inch. As the pressures increased, the yields decreased. Cook 45, with a pressure of 40 pounds per square inch, resulted in a product so much undercooked that no pulp could be prepared from it. The yield, of course, was very high. Cook 46, using a pressure of 140 pounds per square inch, resulted in 50 per cent yield. For intermediate pressures the yields were correspondingly higher.

TABLE 8.—*Effect of varying pressures on the yield of pulp.*

Weight of chips charged (bone-dry basis)	pounds	1.000 to	1.005
Water in chips	per cent	10.4 to	11.0
Causticity of liquor charge	do		83.5
Sulphidity of liquor charge	do		27.4
Initial volume of digester liquors per pound of chips (bone-dry basis)	gallons	0.667 to	0.680
Duration of cooking, total	hours		3.0
Duration of cooking at zero gauge pressure	do		0.1
Duration of cooking at maximum gauge pressure	do	2.0 to	2.3
Total duration of beater treatment (at light brush only)	do	0 or	1

Cook No.	Liquor charge, initial concentrations.			Chemicals charged per 100 pounds of chips (bone-dry basis).			Maximum gauge pressure.	Yield of crude pulp (bone-dry basis).
	NaOH.	Na_2S.[1]	All sodium compounds as Na_2O.	NaOH.	Na_2S.[1]	All sodium compounds as Na_2O.		
	Grams per liter.	*Grams per liter.*	*Grams per liter.*	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Per cent.*
45	21.3	10.6	30.8	12.0	6.0	17.4	40	(2)
42	21.1	10.6	30.6	12.0	6.0	17.4	80	61.3
39	21.2	10.6	30.7	12.0	6.0	17.4	90	60.2
41	21.1	10.6	30.6	12.0	6.0	17.4	120	54.0
46	21.3	10.6	30.8	12.0	6.0	17.4	140	50.0

(P. L.—138, S. L.—3 *b*.)

[1] The same values apply to the Na_2SO_4 used.

[2] Wood not cooked; no pulp prepared.

Pulps produced with the higher pressures were stronger and had better wearing properties than those resulting from the lower pressures. With lower pressures the pulps became more and more brittle and gradually lost their soft, pliable, leather-like feel. The pulps resulting from the lower pressures were the more brown in color.

The best pressure conditions for these tests seemed to be from 100 to 140 pounds per square inch. If larger amounts of chemicals had been employed, pulps of the same yield and properties would probably have resulted from pressures of 80 to 100 pounds per square inch.

EFFECTS OF VARYING THE DURATIONS OF COOKING.

Since the time from the start of a cook until maximum pressure was obtained in the autoclave was practically constant (varying from 0.5 to 0.7 hour), only the total duration of cooking will be considered. Table 9 shows how the yields were affected for total durations varying from one to nine hours in three series of tests, using high, medium, and low amounts of chemicals. In the case of the first series, employing very high amounts of chemicals, 55.9 per cent of the wood (giving a yield of 44.1 per cent) was dissolved during two hours of cooking, while by cooking for seven hours longer an additional loss of only 12.8 per cent occurred. Cook 124, with a total duration of but one hour, afforded the best pulp and the highest yield for this series. This pulp came from the autoclave in the form of soft chips, and the resultant paper made from the beaten pulp was firm and strong, with good resistance to wear. The other pulps were soft and fuzzy, due to overcooking. As the duration increased, the color of the pulps changed from brown (cook 124) to light gray (cook 78).

TABLE 9.—*Effect of varying durations of cooking on the yield of pulp.*

Weight of chips charged (bone-dry basis)	pounds..	0.964 to 1.034
Water in chips	per cent..	16.0 to 24.4
Causticity of liquor charge	do....	51.6 to 53.3
Sulphidity of liquor charge	do....	26.5 to 27.4
Initial volume of digester liquors per pound of chips (bone-dry basis)	gallons..	0.667 to 0.680
Duration of cooking at zero gauge pressure	hours..	0.1
Maximum gauge pressure per square inch	pounds..	90
Total duration of beater treatment (at light brush only)	hours..	1.5 or 2

FIRST SERIES.

Cook No.	Liquor charge, initial concentrations.			Chemicals charged per 100 pounds of chips (bone-dry basis).			Duration of cooking.		Yield of crude pulp (bone-dry basis).
	NaOH.	Na_2S.[1]	All sodium compounds as Na_2O.	NaOH.	Na_2S.[1]	All sodium compounds as Na_2O.	Total.	At maximum gauge pressure.	
	Grams per liter.	*Grams per liter.*	*Grams per liter.*	*Pounds.*	*Pounds.*	*Pounds.*	*Hours.*	*Hours.*	*Per cent.*
124	71.9	36.0	104.7	40.0	20.0	58.3	1.0	0.5	57.4
125	71.9	36.0	104.7	40.0	20.0	58.3	2.0	1.5	44.1
80	72.0	36.0	104.7	40.0	20.0	58.2	5.0	4.3	37.8
78	72.0	36.0	108.1	40.0	20.0	60.1	9.0	8.3	31.3

SECOND SERIES.

123	44.9	22.5	65.4	25.0	12.5	36.4	1.0	0.5	68.6
126	44.9	22.5	65.4	25.0	12.5	36.4	2.0	1.5	48.5
84	45.0	22.7	65.6	25.0	12.6	36.5	5.0	4.3	45.0
83	45.0	22.7	65.6	25.0	12.6	36.5	9.0	8.3	38.2

THIRD SERIES.

86	21.6	10.8	31.4	12.0	6.0	17.4	1.0	0.3	80.9
127	21.6	10.8	31.6	12.0	6.0	17.6	2.0	1.5	66.7
88	21.2	10.6	30.8	12.0	6.0	17.4	5.0	4.3	59.0
87	21.6	10.8	31.4	12.0	6.0	17.4	9.0	8.3	57.0

(P. L.—138, S. L.—176.)

[1] The same values apply to the Na_2SO_4 used.

In the second series, when medium amounts of chemicals were used, prolonging the durations of cooking likewise resulted in decreasing the yields. The yield for cook 123, with a total duration of one hour, was 68.6 per cent; and cook 83, with a total duration of 9 hours, had a yield of 38.2 per cent. With a 2-hours' duration the amount of the wood dissolved was 51.5 per cent (48.5 per cent yield), while with a 7-hours' longer cooking period the loss was only 10.3 per cent additional. The best kraft pulps were obtained from cooks 126 and 84, with total durations of 2 and 5 hours, respectively. The resultant papers were firm and strong, and resistant to wear. Cook 123, using a duration of 1 hour, resulted in a weak, brittle, and undercooked pulp, while the pulp from cook 83, which had a duration of 9 hours, was soft, fluffy, and evidently overcooked.

The same general effects resulted from varying the durations in the third series of tests in which comparatively low amounts of chemicals were employed. In this case, however, the best pulps were produced with the longer durations, 5 hours for cook 88 and 9 hours for cook 87. The tests employing shorter durations resulted in weak and brittle pulps, due to undercooking. The pulp from cook 88 was slightly inferior to that from cook 87, but both would be considered of fair quality for making kraft wrapping paper.

The results from the three series of tests indicate that cooks employing high amounts of chemicals and very short durations will afford pulps of a quality and yield similar to those obtained when using medium amounts of chemicals and medium durations and to those resulting from the use of low amounts of chemicals and comparatively long durations. It is evident, however, that much more careful control of the operations must be exercised in order to obtain consistent results when high amounts of chemicals are employed.

EFFECTS OF VARYING THE INITIAL CONCENTRATIONS.

In each of two series of tests varying the initial concentrations of chemicals in the liquor charge the amounts of chemicals per 100 pounds of wood were held constant as follows: 15 pounds of caustic soda, 7.5 pounds of sodium sulphide, and 7.5 pounds of sodium sulphate for the first series, and 12 pounds of caustic soda, 6 pounds of sodium sulphide, and 6 pounds of sodium sulphate, for the second series. Since the amounts of chemicals were held constant, and the concentrations varied, the initial volumes of digestor liquors per pound of chips also varied accordingly. Table 10 shows the effect of the varying concentrations on the yield of pulp.

TABLE 10.—*Effect of varying initial concentrations on the yield of pulp.*

Weight of chips charged (bone-dry basis)	pounds..	0.964 to 1.031	
Water in chips	per cent..	16.4 to 24.4	
Causticity of liquor charge	do....	52.8 to 53.6	
Sulphidity of liquor charge	do....	27.2 to 27.5	
Duration of cooking, total	hours..	3.0	
Duration of cooking, at zero gauge pressure	do....	0.1	
Duration of cooking at maximum gauge pressure	do....	2.0 to 2.5	
Maximum gauge pressure per square inch	pounds..	90	
Total duration of beater treatment (at light brush only)	hours..	2	
Chemicals charged per 100 pounds of chips (bone-dry basis):		*First series.*	*Second series.*
NaOH	pounds..	15.0	12.0
Na_2S	do....	7.5	6.0
Na_2SO_4	do....	7.5	6.0
All sodium compounds as Na_2O	do....	21.8	17.5

FIRST SERIES.

Cook No.	Liquor charge, initial concentrations. NaOH.	Na_2S.	Na_2SO_4.	All sodium compounds as Na_2O.	Initial volume of digester liquors per pound of chips (bone-dry basis).	Yield of crude pulp (bone-dry basis).
	Grams per liter.	*Grams per liter.*	*Grams per liter.*	*Grams per liter.*	*Gallons.*	*Per cent.*
89	60.0	30.0	30.0	87.2	0.300	47.9
90	45.0	22.5	22.5	65.1	.400	53.3
91	36.0	18.0	18.0	52.1	.500	55.2
93	30.0	15.0	15.0	43.6	.600	58.6
94	25.7	12.9	12.9	37.4	.700	61.3
95	22.5	11.2	11.2	32.7	.800	64.4
96	20.0	10.0	10.0	29.0	.900	66.4
97	18.0	9.0	9.0	26.1	1.000	66.9

SECOND SERIES.

112	72.0	36.0	36.0	104.9	0.200	51.0
100	49.7	24.8	24.8	72.0	.290	51.1
101	36.0	18.0	18.0	52.2	.400	52.3
105	28.8	14.6	14.4	42.2	.500	56.0
114	24.0	12.0	12.0	35.0	.600	62.6
106	20.6	10.3	10.3	30.0	.700	60.6
107	18.0	9.0	9.0	26.2	.800	66.0
108	16.0	8.0	8.0	23.3	.900	67.4
115	14.4	7.2	7.2	21.0	1.000	67.3
110	12.0	6.0	6.0	17.5	1.200	67.8
111	10.3	5.1	5.1	15.0	1.400	67.4

(P. L.—138, S. L.—176.)

When the concentration of all sodium chemicals expressed as Na_2O was varied from 26.1 to 87.2 grams per liter (first series of tests) the resultant yield decreased from 66.9 to 47.9 per cent. The best results, considering both yield and quality of pulps, were obtained from cooks 91 and 93, using Na_2O concentrations of 52.1 and 43.6 grams per liter, respectively. Pulps produced from cooks having lower concentrations were brittle and lacked strength and wearing properties. In the second series of tests, using somewhat smaller amounts of chemicals, the higher concentrations afforded the better results. The best pulp with regard to strength and wearing properties was that obtained from cook 112, using a Na_2O concentration of 104.9 grams per liter. The pulps obtained when using a concentration of 35 grams per liter or less were quite brittle, and had little strength and poor wearing properties.

SUMMARY OF CONCLUSIONS FROM THE AUTOCLAVE TESTS.

(1) The effective cooking chemicals in sulphate cooking liquors are caustic soda and sodium sulphide, the former being the more drastic in its action. Sodium sulphate and sodium carbonate, which unavoidably occur in the commercial liquors, are of no assistance in cooking, at least so far as the wood of longleaf pine is concerned.

(2) Increases in the amounts of either caustic soda or sodium sulphide, or both, result in more thorough cooking. The same effect may be obtained by increasing either the cooking pressure, the duration of cooking, or the initial concentrations of the chemicals in the cooking liquors.

(3) More thorough cooking is evidenced by decreased yields and by lighter colored pulps until a condition of very thorough cooking is reached, after which the color of the pulp is not affected.

(4) The best, or well-cooked, sulphate kraft pulps will have good strength and wearing properties, will be light brown in color, and will have a smooth, firm, leather-like feel when properly beaten. Undercooked pulps are characterized by a darker brown color, brittleness, lack of strength, and poor wearing properties. Overcooked pulps are light gray in color and may have good strength and wearing properties when properly beaten, but the yield will be low. Pulps much overcooked, in addition to being light gray in color, will be soft and fluffy, with little strength.

(5) With each different combination of the cooking conditions there is a definite minimum amount of sodium sulphide which must be used in conjunction with the caustic soda present to impart to the product the high strength and good wearing properties characteristic of properly cooked sulphate kraft pulps.

(6) The use of sodium chloride in conjunction with caustic soda improves the quality of the pulp to a slight extent only. The similar use of sulphur results in pulps having properties practically the same as those of sulphate pulps.

(7) As the proportion of sodium sulphide in the digester charge is increased, the disagreeable odor produced in the cooking operations becomes more pronounced.

PRACTICAL SIGNIFICANCE OF THE EXPERIMENTS.

While the present experiments are not complete, they show conclusively (1) that longleaf pine is well adapted for the manufacture of natural-color kraft pulps and papers; (2) that the sulphate process of pulp making applied to this wood affords products of better quality and of higher yields than the soda process; (3) that kraft papers can be made from longleaf pine equal or superior in quality to the imported and domestic kraft papers now on the market; and (4)

that the high gravity of the wood and the resultant high yield of pulp per cord give longleaf pine an advantage possessed by few, if any, other commercially important woods suitable for pulp making.

The autoclave tests indicate that there should be a certain combination of values for the variable cooking conditions which will result in the most economical method of operation. However, other factors than the variables thus far investigated must be taken into consideration in determining what this combination is. For example, the proper degree to which a pulp must be cooked will depend partly upon the cost of the beater treatment. With cheap power for beating, the pulp need not be so severely cooked as when the cost of power is high. The best concentrations and proportions of chemicals in the digester liquors will likewise depend upon the efficiency of the recovery system and the method of operating it.

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